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Effect of annealing on microstructure and properties of Er modified 5052 alloy

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ABSTRACT

Microstructure of 5052 + Er alloy at continuous casting, hot-rolled, cold-rolled and annealed state were observed, studied the effect of annealing on the microstructure, mechanical properties and forming properties of the alloy, and explored the mechanism of its action. The results showed that, when the annealing temperature is between 340 °C and 360 °C, the tensile strength and yield strength of the 5052 + Er alloy have a large decline while the elongation increase obviously; The recrystallization starting temperature of the alloy is 320 °C, and recrystallization ending temperature is 320 °C; The average strength ratio of the 5052 + Er alloy with 360 °C annealing for 2 h is 0.592, when the average strain hardening exponent is 0.3882, which shows the alloy has good ductility and formability; the second Al3Er phase with the size about 20–30 nm has the coherent/semi coherent relationship with the matrix, which can play an inhibitory role of recrystallization.

Introduction

As one of the most extensive used 5 × × × series aluminum alloy, 5052 alloy was widely used in construction, packaging, lamp bracket and rivets, metal products, electrical enclosures etc [1] because of its good forming processing performance, corrosion resistance, weldability and moderate intensity. The main alloy element of the alloy is magnesium with content between 2.2 and 2.8%, as a representative of the non heat treatment strengthening Al-Mg alloy, work hardening was usually used to increase its strength, However, there are many defects such as dislocations and vacancies in the alloy after rolling deformation, which will lead to the instability of the alloy, subsequent annealing treatment can induce the deformation of the alloy in the process of recovery and recrystallization process so that the effect of hardening is reduced [2]. For building aluminum alloys, because of its special application environment, requires higher strength and more excellent forming properties, etc than the ordinary aluminum plate, thus the application range and the safety reserve capacity of the building aluminum profile can be improved. The method of adding a certain amount of trace element in the Al-Mg alloy can improve the strength of the alloy, and also play a stabilizing role in the properties of the alloy. Previous studies showed that rare earth elements Sc, Er etc can form nanoscale second phase particles with $L1_2$ structure, which can refine the grain size, improve the recrystallization temperature and the strength of the alloy [3]. However, until now, there is little research on the rolling deformation and annealing behavior of Er modified 5052 alloy, and the mechanism of its action is not clear. This paper starts from the continuous casting and rolling 5052 + Er alloy slab, studied the effect of different annealing temperature on the mechanical properties, forming properties and microstructure of 5052 + Er alloy, it provides the necessary reference for the development and application of high performance 5052 alloy.

Materials and methods

Preparation of 5052 + Er alloy sheet by high flux continuous casting and rolling process, specific processes include: batching, smelting, continuous casting (cast billet with 20 mm), hot rolling (to 4.5 mm) and multi pass cold rolling (to 2.0 mm), and other steps. The chemical composition of 5052 + Er alloy was measured by inductively coupled plasma atomic emission spectrometry, and the main element mass fraction (%) was: 2.60Mg, 0.11Mn, 0.08Cu, 0.14Si, 0.09Er, 0.12Fe, and the remainder was Al. The cold rolled sheets were annealed in the box type resistance furnace, with annealing temperature between 220 °C and 440 °C, and the annealing time is 2.5 h.

Metallographic samples were cut by wire cutting method, and followed by different types of sandpaper grinding step by step, mechanical polishing and corrosion in 0.5% HF reagent, the microstructure

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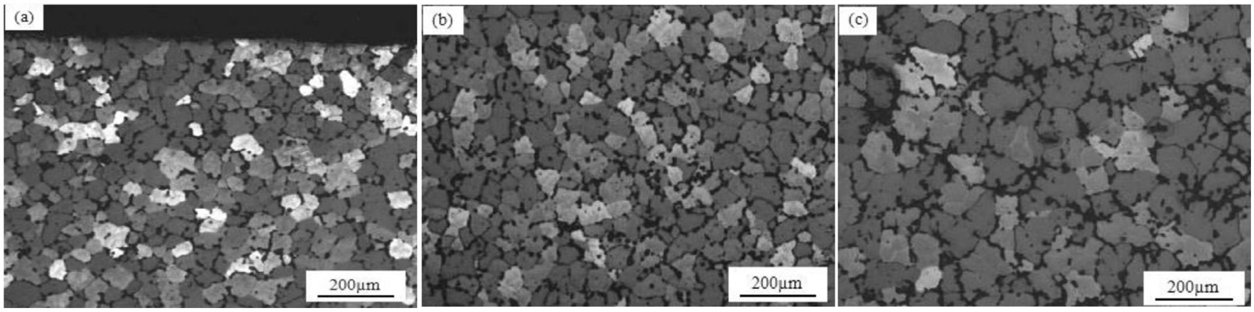

Fig. 1. Optical microstructure of 5052 + Er alloy (a) upper surface (b) lower surface (c) middle.

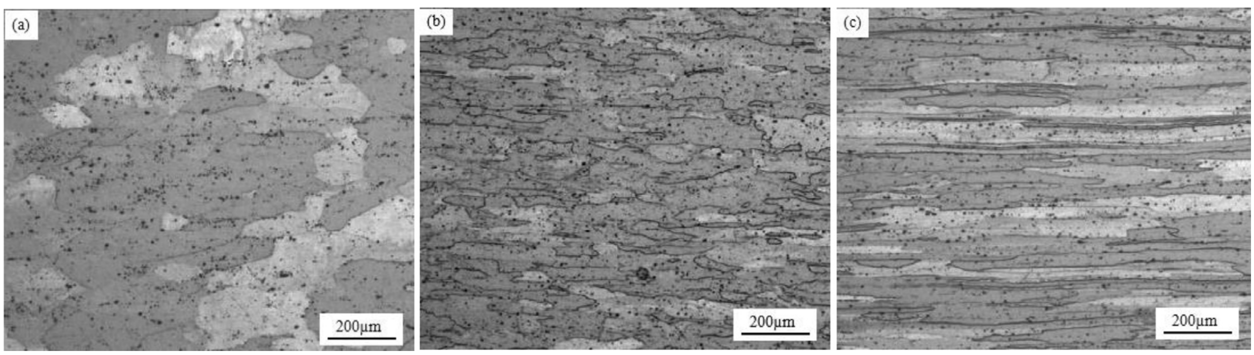

Fig. 2. Optical microstructure of hot rolled alloy (a) surface (b) cross section (c) longitudinal section.

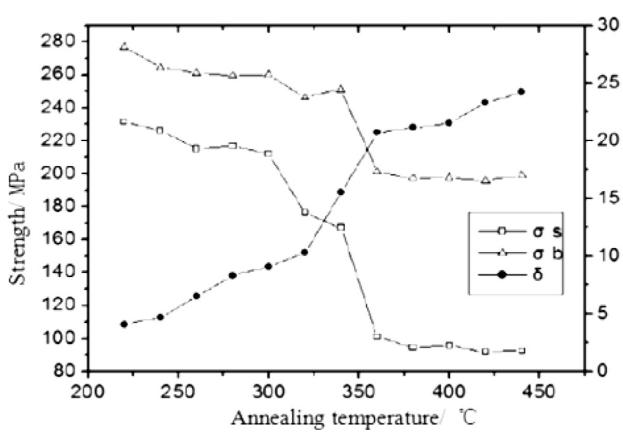

Fig. 3. Mechanical properties and annealing temperature curve of 5052 + Er alloy.

observation was carried out on the Zeiss Axio Lab.A1 type optical microscope; Mechanical testing in the United States MTS-810 hydraulic servo electronic universal tensile testing machine, with the tensile rate is 1.5 mm/min; The strain hardening index was determined by reference to the standard GB/T 5028-2008 "Determination of tensile strain hardening index of sheet metal and thin strip" method; Microhardness test was carried out in the Vivtorinox HVS-1000 digital hardness tester with the load is 200 g and the holding time is 10 s; TEM specimen were mechanical thinning to 30–70 μm, and then double perforation with 25% nitric acid methanol solution (temperature control below −35 °C), which were observed on JEOL-2010 transmission electron microscope.

Results and analysis

The microstructure of upper surface, lower surface and middle on 5052 + Er alloy slab by continuous casting was observed, and the results were shown in Fig. 1, As can be seen, the upper and lower surfaces of the 5052 + Er alloy slab are fine grain zones, the average grain size of the upper surface is 70.74 μm, and the average grain size of the lower surface is 70.28 μm, the grain size of the upper and lower surfaces is considerable. In the middle of 5052 + Er alloy slab is coarse equiaxed grains, the average grain size is 93.08 μm. In addition, no obvious dendrite structure and other metallurgical defects exist in the grain interior of the upper surface, the lower surface or the middle part.

Fig. 2 shows the optical microstructure of the surface, cross section and longitudinal section of hot rolled 5052 + Er alloy plate with the thickness about 4.5 mm. It can be seen that the surface of the hot rolled slab of 5052 + Er alloy appears to be a certain number of recrystallization grains, and the microstructure of the cross section and longitudinal section can be seen in strip shape along the rolling direction.

Annealing treatment of 5052 + Er alloy plate after cold rolled was carried out, the mechanical properties of 5052 + Er alloy at room temperature with different annealing temperatures are shown in Fig. 3, in which σs and σb denote yield strength and tensile strength respectively, andδdenote elongation. With increasing annealing temperature, the tensile strength and yield strength of the alloy showed a trend of gradually decreased, and elongation increased gradually (The tensile strength of alloy annealed at 340 °C is slightly greater than 320 °C,

Table 1
Mechanical properties of 5052 + Er alloy after annealing at 360 °C/2 h.

Performance index	Sample-1	Sample-2	Sample-3	Sample-4	Sample-5	Average value
Yield strength ratio	0.588	0.596	0.594	0.590	0.592	0.592
Strain hardening exponent (n Value)	0.3881	0.3884	0.3885	0.3880	0.3879	0.3882

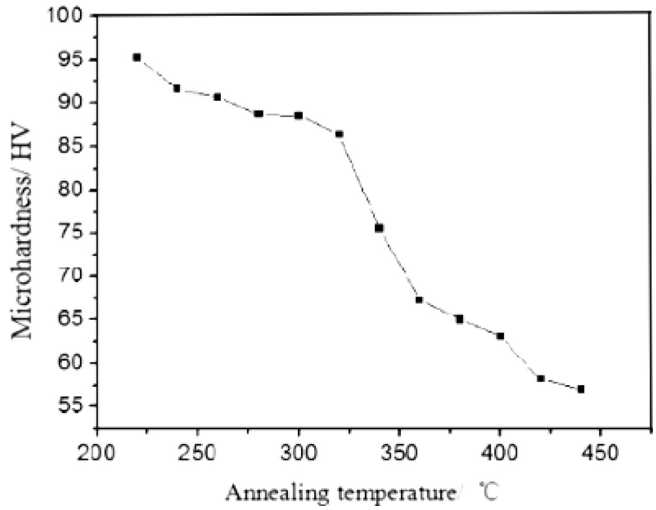

Fig. 4. Relationship of micro hardness and annealing temperature of 5052 + Er alloy.

which may be due to the error of the measurement, but does not change the trend). The annealing process can be divided into three stages according to the mechanical properties of 5052 + Er alloy with different annealing temperature: the first stage as the annealing temperature at 220 °C–320 °C, in this stage, the tensile strength and yield strength decreased slowly, and elongation increased slowly; the second stage as the annealing temperature at 340 °C–360 °C, the tensile strength and yield strength decreased rapidly, but its elongation increases rapidly; the third stage as the annealing temperature at 380 °C–440 °C, the change of tensile strength, yield strength and elongation of the alloy tends to be slow. This is mainly due to the cold rolled sheet has experienced a large plastic deformation, a large number of deformation energy storage, recovery and recrystallization can be occurred during annealing process, and recrystallization behavior will offset the deformation in the process of hardening [4], which reduces the strength and increase the plasticity.

The yield strength ratio and strain hardening exponent of the 5052 + Er alloy plate annealed at 360 °C/2 h were statistical analyzed, the results are shown in Table 1. The yield strength ratio is the ratio of the yield point (yield strength) and tensile strength, the lower yield strength ratio indicates that the material has better plasticity, the higher yield strength ratio indicates that the material has better anti deformation ability; Strain hardening exponent n represents homogeneous deformation ability, the larger the value of n, the stronger the uniform deformation ability of the alloy during plastic deformation, the alloy would not prone to rupture in the deformation process [3]. From the point of view of mechanics performance test results of the 5 groups of parallel samples, the average yield strength ratio of 5052 + Er alloy is 0.592, and the average strain hardening exponent is 0.3882 respectively, the former index indicates that the alloy has good plasticity, while the latter showed that the alloy has good forming performance, which can meet the requirements of complex shape of the alloy on the

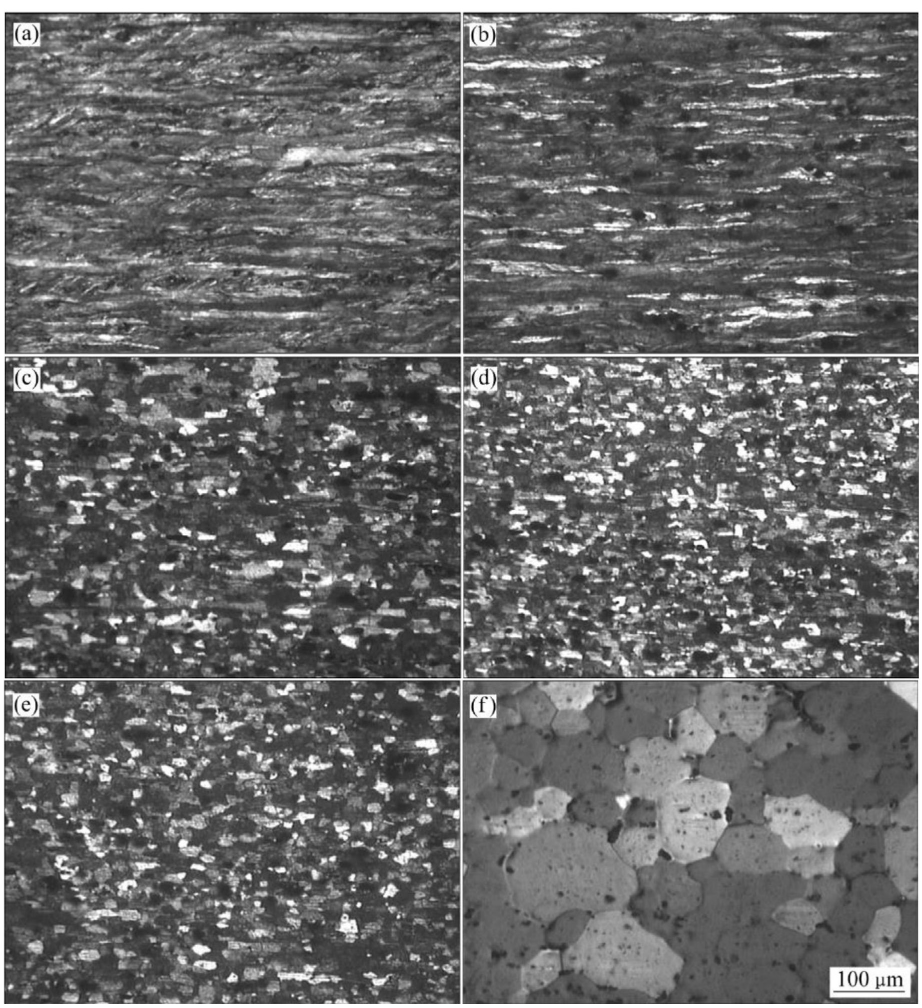

Fig. 5. Microstructure of 5052 + Er alloy at different annealing temperature. Cold-rolled; Annealing: (b) 220 °C; (c) 320 °C; (d) 360 °C; (e) 400 °C; (f) 440 °C.

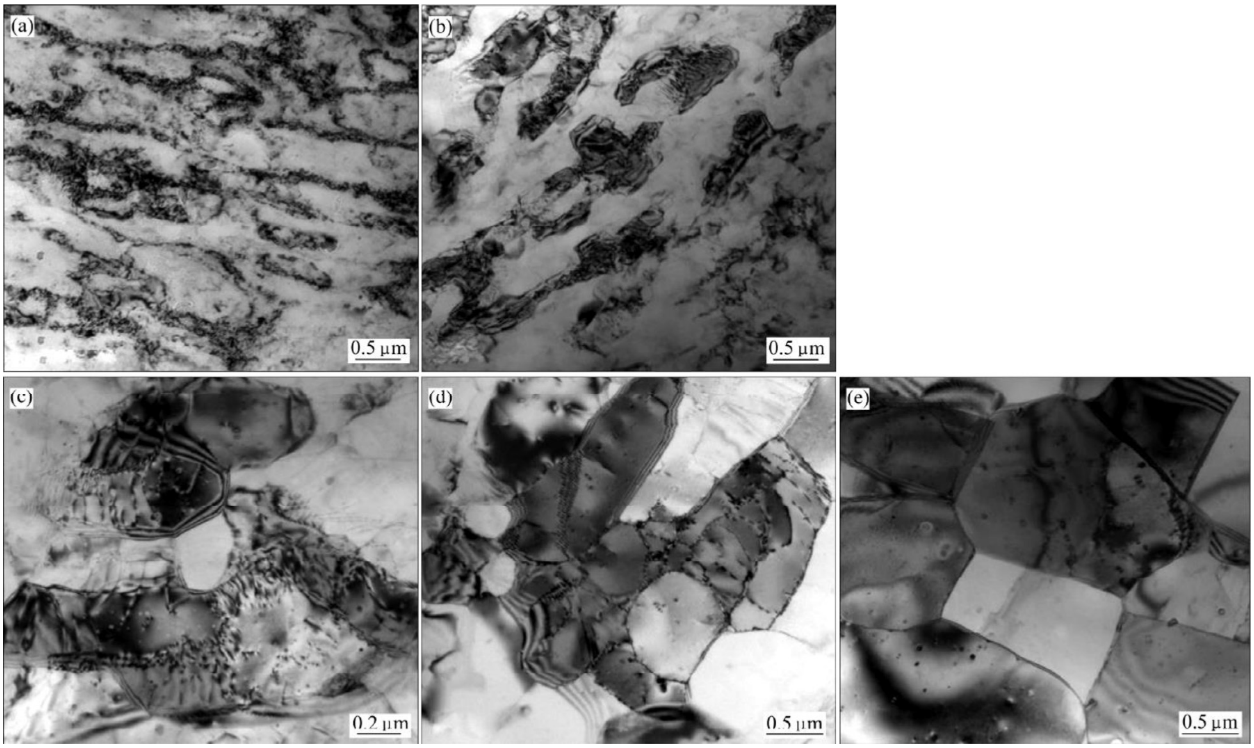

Fig. 6. TEM morphology of 5052 + Er alloy at different annealing temperature. (a) Cold-rolled; Annealing: (b) 220 °C; (c) 320 °C; (d) 360 °C; (e) 400 °C; (f) 440 °C.

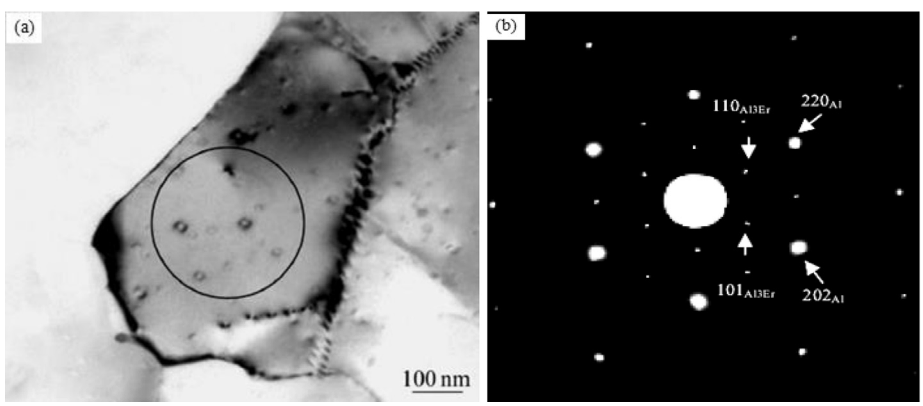

Fig. 7. (a) TEM and (b) selected area electron diffraction patterns of the second phase particles after annealing at 360 °C for 2.5 h.

surface quality and strong plastic.

Fig. 4 shows the variation of microhardness of 5052 + Er alloy under different annealing temperatures. The microhardness decreased slowly when the annealing temperature at 220 °C–320 °C, and the microhardness decreased faster when the annealing temperature at 320 °C–360 °C; when the annealing temperature of more than 360 °C, the microhardness decreased to a lesser extent and tends to be stable. Thus it can be seen that the recrystallization starting temperature of 5052 + Er alloy is 320 °C, while the recrystallization ending temperature is 360 °C. When the annealing temperature below the recrystallization starting temperature, the recovery process of internal stress relief in the alloy has been eliminated, and when the annealing temperature reaches the recrystallization temperature, the alloy begins to form recrystallization structure; continue increasing annealing temperature will cause the recrystallization grain coarsening and growth, Reflected in the mechanical properties are the decline in hardness, the strength decreased and the plasticity increased [5].

Fig. 5 shows the microstructure of cold rolled and annealed 5052 + Er alloys with different temperature. For the 5052 + Er alloy with cold-rolled and annealed at 220 °C, the microstructure is still fiber shaped rolling deformation; when the annealing temperature rise to 320 °C, in addition to fibrous microstructure, the alloy can also find partial recrystallization; to elevated annealing temperature to 360 °C, this alloy has undergone a complete recrystallization, forming a fine recrystallized grains, and fibrous microstructure completely disappeared; annealing temperature continue to rise to 400 °C, there is no obvious coarsening for fine recrystallized grains, but when the annealing temperature increased to 440 °C, the alloy has occurred obvious recrystallization grain growth phenomenon. The microstructure of the 5052 + Er alloy at different annealing temperatures was observed and the results were consistent with the previous mechanical properties and hardness test results, namely when the annealing temperature of 320 °C, the 5052 + Er alloy began to recrystallization, and when the annealing temperature increased to 360 °C, the alloy has basically completed the recrystallization; if continue to increasing annealing temperature, some grains will appear irregular growth and into recrystallization grain coarsening and growth stage [6].

Fig. 6 shows the TEM microstructure of cold rolled and annealed

5052 + Er alloys with different temperature. After multi pass cold rolling treatment, the grain was elongated and compressed along the rolling direction at cold rolled state, dislocations formed by high density dislocations are distributed in the alloy; After annealing treatment of 5052 + Er alloy with 220 °C for 2.5 h, the dislocation density in the alloy decreased, and in the local area can be seen obviously cellular sub structure, there are a lot of regular array of dislocations in the cell wall, which is typical of the recovery process [7]; continue to increase of the annealing temperature to 320 °C, dislocation cell sub structure disappeared and the cell wall is thin, the alloy has undergone recrystallization, sub grain structure and the existence of the second phase particles in local area are visible; further increase the annealing temperature to 360 °C, the alloy can be found subgrain structure obviously, the recrystallization nucleation has been formed by merging and growing up, which shows the recrystallization has been carried out more fully; when the annealing temperature rises to 440 °C, recrystallization grain occurs to a certain degree of coarsening and growth.

The second phase particles in 5052 + Er alloy annealed at 360 °C for 2.5 h was observed by TEM, the results are shown in Fig. 7. The bean shaped second phase particles with the size of about 20–30 nm can be found in sub grain structure, the average spacing between the individual particles are about 0.07 μm, selected area electron diffraction combined with literature analysis [8] showed that, the second phase particles were Al_3Er phase which shows the coherent / semi coherent relationship with the matrix, it can play a role on pin the grain boundaries and inhibited the subgrain boundaries merging and migration, to a certain extent, the structure of subgrain is stabilized and the recrystallization of the alloy is suppressed [9].At a certain temperature, the average size of grain has the relationship with time as follow [10]: $D = Bt^n$, In which, D is the grain size, B is the constant relative to time, n will be influenced by the second phase in the alloy, dispersive second phases can strongly pin dislocation and subboundaries, hinder their climb and increase the shear stress required by glide of dislocation, recrystallization is strongly restrained and the high strength caused by cold hardening is preserved. Thus, the value of n will decrease. In this way, the average size of grain will refined.

These fine dispersed Al_3Er particles can strongly pin up the dislocations and subboundaries, which can make substructure stable. In the process of recovery before recrystallization, they prevent the movement of dislocations and the incorporation of subboundaries, and keep a rather high dislocation density, thus delayed the beginning of the recrystallized nucleation. During the subsequent growth of the nucleus, the stable substructure makes it difficult for the migration of crystal boundaries, thus inhibit the growth of the recrystallized nucleation. As a result, the recrystallization can be prevented and the recrystallization temperature shall be increased. If recrystallization is produced, the equilibrium Al_3Er phase still keeps up with the matrix. Therefore continuous recrystallization is inhibited by the Al_3Er phase in the deforming process at high temperatures, thus the grain growth is inhibited. It plays an important role for stabilizing deformation structure at high temperatures, the fine precipitation Al_3Er phase can gather and grow only in the high temperature, which makes the distance between particles become large, thus the pining effect on dislocation and subboundaries can be reduced, and the recrystallization process is accelerate.

Conclusions

1) The average grain size of the upper surface, bottom surface and the middle region on 5052 + Er alloy slab are 70.74 μm, 70.28 μm and 93.08 μm respectively, the grain interior have no obvious characteristics of the dendritic structure; the surface of 5052 + Er alloy hot rolled slab appeared a certain number of recrystallization grains, and the grain in the cross-section and longitudinal section were flattened and elongated along the rolling direction.
2) With the increase of annealing temperature, the tensile strength and yield strength of the 5052 + Er alloy showed a trend of decreased, and the elongation increased gradually; When the annealing temperature at 340 °C–360 °C, the tensile strength and yield strength of 5052 + Er alloy showed larger decline, while the elongation increased obviously.
3) Recrystallization starting temperature of 5052 + Er alloy is 320 °C, while the recrystallization ending temperature is 360 °C; the second Al_3Er phase with the size about 20–30 nm has the coherent/semi coherent relationship with the matrix, which can play an inhibitory role of recrystallization.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.rinp.2018.06.048.

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